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(71) Applicant: BUSH BOAKE ALLEN INC. [US/US]; 7 Mercedes Drive, Montvale, NJ 07645 (US).

(72) Inventors: CHALUPA, William, F.; 3 Matthew Drive, Chestnut Ridge, NY 10977 (US). CALZOLARI, Loris; 1558A Davenport Road, Toronto, Ontario M6H 2J1 (CA).

(74) Agent: MUCCINO, Richard, R.; 758 Springfield Avenue, Summit, NJ 07901 (US).

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(54) Title: METHOD OF ENCAPSULATION AND PRODUCT PRODUCED THEREBY

(57) Abstract

The present invention is directed to an encapsulated material which comprises (a) a core comprising an encapsulatable material; and (b) a coating layer over the core comprising a protein having a mixture of hydrophobic and hydrophilic properties selected from the group consisting of isolated soy protein, whey protein isolate, caseinate, and mixtures thereof. The present invention is also directed to a method for encapsulating an encapsulatable material with a protein having a mixture of hydrophobic and hydrophilic properties which comprises the steps (a) preparing an aqueous mixture of a protein having a mixture of hydrophobic and hydrophilic properties selected from the group consisting of isolated soy protein, whey protein isolate, caseinate, and mixtures thereof; (b) adding an encapsulatable material to the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (a); and (c) denaturing the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (b) to precipitate the protein and thereby encapsulate the encapsulatable material in the protein. The present invention is further directed to the novel encapsulated material prepared by the novel process.

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METHOD OF ENCAPSULATION AND PRODUCT PRODUCED THEREBY

This application is a continuation-in-part application of application serial no. 08/666,698, filed 18 June 1996.

FIELD OF THE INVENTION

The present invention is directed to an encapsulated material which comprises (a) a core comprising an encapsulatable material; and (b) a coating layer over the core comprising a protein having a mixture of hydrophobic and hydrophilic properties selected from the group consisting of isolated soy protein, whey protein isolate, caseinate, and mixtures thereof. The present invention is also directed to a method for encapsulating an encapsulatable material with a protein having a mixture of hydrophobic and hydrophilic properties which comprises the steps of (a) preparing an aqueous mixture of a protein having a mixture of hydrophobic and hydrophilic properties selected from the group consisting of isolated soy protein, whey protein isolate, caseinate, and mixtures thereof; (b) adding an encapsulatable material to the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (a); and (c) denaturing the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (b) to precipitate the protein and thereby encapsulate the encapsulatable material in the protein. The present invention is further directed to the novel encapsulated material prepared by the novel process.

DESCRIPTION OF THE BACKGROUND

For certain applications in aqueous media, it is desirable to convert a water-soluble color into a water-insoluble color. For example, lake colors are water-soluble colors that have been precipitated and immobilized on an aluminum hydroxide support and consequently behave like water-insoluble colors. However, when lake colors are used in acidic solutions, the acidic solution reacts with and dissolves the alumina hydroxide support thereby liberating the water-soluble color and permitting it to leach into the acidic solution. This acid labile characteristic of lake colors is undesirable because the distinguishable color differentiation between the lake color and the acidic solution is lost.

United States patent application serial no. 08/383,038, filed 3 February 1995, and entitled "A Colored Jelly-Type Substance, Method of Preparation, and Composition Containing Same" discloses and claims water-insoluble colors to color jelled substances. The colored jelled substances can be incorporated into acidic liquid compositions, such as beverages, without loss of color differentiation.

However, not all colors are available as water-insoluble colors. For example, there are no food grade water-insoluble blues or water-insoluble greens approved in the United States. Accordingly, it would be desirable to provide a means to convert a water-soluble color into a water-insoluble color.

United States Patent no. 5,601,760 (*Rosenberg*) discloses a method for microencapsulation of a volatile or non-volatile core material in a wall system consisting essentially of a whey protein. The method comprises the steps of (a) preparing the wall system using whey protein; (b) mixing a core system within the wall system; (c) dispersing the core system in the wall system to form an emulsion, dispersion or a combination thereof; (d) microencapsulating the emulsion, dispersion or the combination of step (c); and (e) harvesting a microencapsulated product. *Rosenberg* states that the wall system formed by microencapsulation protects the core against deterioration by oxygen, or from detrimental effects of other compounds or materials, limits the evaporation or losses of volatile core materials, and releases the core upon full hydration reconstitution. The whey protein is selected from the group consisting of a whey protein isolate, a whey protein concentrate, ß-

lactoglobulin, α-lactalbumin, a mixture thereof, a modified or denatured whey protein and a fraction thereof. Examples of the core are fats, such as anhydrous milkfat, volatiles, essential oils flavors, fragrances, nutritional compounds, health products, vitamins, oleoresins, bacteria, enzyme, minerals, natural colorants, oils, essences, pharmaceuticals, and pharmaceutically acceptable ingredient, or a mixture thereof. *Rosenberg* states that the most used, practical and preferred process for microencapsulation of volatile or non-volatile ingredients according to this invention is spray-drying.

United States Patent no. 5,051,304 (*David et al.*) discloses a process for preparing microcapsules containing an encapsulated substance comprising a hydrophobic liquid or a water-insoluble solid. The process comprises the steps of (a) emulsifying a hydrophobic liquid or suspending a water-insoluble solid in a gelatin solution, (b) adding a low molecular weight hydrosoluble anionic polysaccharide to the gelatin solution, and (c) encapsulating the hydrophobic liquid or water-insoluble solid within microcapsules by complex coacervation of the gelatin and polysaccharide. *David et al.* states that the polysaccharides are generally not found naturally and are obtained by physical, chemical, or enzymatic depolymerization of the natural anionic polysaccharides, which are water-soluble or rendered water-soluble. The polysaccharides may be selected from alginates, extracted from brown algae, carrageenan, of the lambda, iota or kappa type, extracted from red algae, pectins extracted from lemons, apples or beetroot, pectates, which result from the demethylation of pectins, or carboxymethylcelluloses, carboxymethylguars or carboxymethyl starches.

United States Patent no. 4,880,649 (*Holzner et al.*) discloses a process for flavoring a composition such as tea, camomile, verbena, or mint in the form of leaves, powders, or particles. The process comprises spraying the composition with an aqueous emulsion comprising 5 to 30% of a solid film-building vehicle selected from the group consisting of polyvinyl acetate, polyvinyl alcohol, dextrins, natural or modified starches, natural or modified proteins, vegetable gums, pectins, xanthanes, carboxymethyl cellulose, methyl cellulose, hydroxyethyl cellulose, and lipoheterosaccharides; 0.1 to 20% of an emulsifying agent selected from the group consisting of fatty-acid mono- or di-glycerides, esters derived from a combination of fatty acids with sorbitol or a saccharide or alkoxylated derivatives thereof, or an ester of tartaric, citric, ascorbic, or lactic acid; and 0.5 to 20% of a non water miscible active volatile flavoring substance. The emulsion-sprayed composition is then dried so that the

flavoring substance is in direct contact with the leaves, powders, or particles of the composition.

United States Patent no. 4,230,687 (*Sair et al.*) discloses a method of encapsulating an active agent as a microdispersion in a storage-stable enveloping matrix of an encapsulating material selected from the group consisting of chemically modified starches, gums, proteins, and mixtures thereof. The method comprises combining the encapsulating material with the active agent and water in an amount limited to ensure maintenance of a homogeneous, high-viscosity paste system during processing. Shearing stress, mechanical working, and heat are applied to the paste system to distribute the active agent throughout the encapsulating material as a microdispersion under processing conditions to avoid compaction and molding compression forces. The encapsulating material may be a protein such as casein, gelatin, or wheat gluten.

United States Patent no. 3,819,838 (*Smith et al.*) discloses a particulate composition comprising multiple capsules each consisting of at least one primary capsule containing a flavoring composition containing volatile components which are released during cooking and baking to flavor the product. The favoring composition consists essentially of a flavoring essence selected from the group consisting of essential oils, oleoresins, and mixtures thereof, encapsulated in a water-soluble encapsulating material selected from the group consisting of natural and modified gums, natural and modified starch, alginates, and proteins. The primary capsules are re-encapsulated in a water-insoluble encapsulating material selected from the group consisting of hydrogenated vegetable oil, fatty acids, fatty esters, and glycerides. The water-soluble encapsulating proteins may be gelatin or casein.

United States Patent no. 3,666,678 (*Mosier et al.*) discloses a process of microencapsulation by coacervation. The method comprises forming a mixture of two liquid phases, one of which is an aqueous solution of a single macrocolloid and the other of which is a partially water-immiscible organic solvent having an organic basic nitrogen compound selected from the class consisting of amines and quaternaries dispersed therein. The macrocolloid is an acid-precursor gelatin soluble in water at both alkaline and acid pH and the aqueous gelatin phase is initially at an alkaline pH. The aqueous gelatin phase is then acidified and the phases intermixed to disperse and emulsify the organic solvent phase in the aqueous phase, thereby providing minute droplets of the

organic solvent containing basic nitrogen compound surrounded by the aqueous solution of the acid-precursor gelatin.

United States Patent no. 3,351,531 (*Noznick et al.*) discloses an encapsulated, dried product comprising 1 to 20% of an enveloping wheat gluten film encapsulating an oil in water dispersion. A quantity of 30 to 70% of an oil or fat is a carrier for 1 to 20% of a material selected from the group consisting of fat soluble food, medicaments, flavoring agents, and food coloring agents.

United States Patent no. 3,116,206 (*Brynko et al.*) discloses a process for the manufacture of minute capsules in a non-gellable film-forming material soluble in water at room temperature. The capsules consist of core materials retained in zein. The process comprises (a) forming an aqueous solution of zein at a pH of approximately 11; (b) dispersing the core materials in the solution; (c) lowering the pH of the solution to below 5.5 to phase-separate the zein around each core as a solvated solid wall; and (d) cross-linking the wall material to form a rigid water-insoluble wall material by treating the capsules with a cross-linking material.

United States Patent no. 2,754,215 (*Evans et al.*) discloses a method for stabilizing the flavor of a volatile essential oil for use in dry food products. The method comprises emulsifying an essential oil in a mixture consisting essentially of an edible non-aqueous water soluble liquid carrier in the presence of an edible non-aqueous protein such as non-fat milk solids and soya flour. Oxidation of reactive aldehydes and terpene polymerization is thereby prevented in storage.

United States Patent no. 5,417,990 (*Soedjak et al.*) discloses a multi-component gelled product having adjacent components of differing colors. At least one of the components contains a water-soluble colorant and either a protein material that produces a relative fluorescence intensity with a standardized ANS test of at least 100 or a polyamino acid. The protein material or polyamino acid is present in the colorant-containing gel component at a level effective to form a stable, water-soluble complex with the colorant. The complex is resistant to migration within the gel layer. Suitable protein materials are alcohol dehydrogenase, serum albumin. egg protein, water-soluble fractions of wheat gluten, and water-soluble fractions of casein. High charge density polyamino acids also function as complexing agents such as polyarginine, polylysine, and polyhistidine.

P. Hongsprabhas et al. [Journal of Food Science, 62: 382-385 (1997)] discloses that increasing the whey protein concentration decreases gel opacity but increases gel strength and water-holding capacity. Increasing the $CaCl_2$ concentration increases gel opacity and gel strength at high protein concentration, but lowers gel strength at protein concentration <10%. Young's modulus and distance to fracture values indicated that gels were most rigid at 30 $mMCaCl_2$, at which point the extent of aggregation (measured by turbidity) was the highest. Increasing $CaCl_2$ concentration from 5 to 150 mM slightly affected the water-holding capacity of Ca^2+-induced gels. Protein concentration was the major factor in determining fracture properties and water-holding capacity.

SUMMARY OF THE INVENTION

The present invention is directed to an encapsulated material which comprises:

(a) a core comprising an encapsulatable material; and

(b) a coating layer over the core comprising a protein having a mixture of hydrophobic and hydrophilic properties selected from the group consisting of isolated soy protein, whey protein isolate, caseinate, and mixtures thereof.

The present invention is also directed to a method for encapsulating an encapsulatable material with a protein having a mixture of hydrophobic and hydrophilic properties which comprises the steps of:

(a) preparing an aqueous mixture of a protein having a mixture of hydrophobic and hydrophilic properties selected from the group consisting of isolated soy protein, whey protein isolate, caseinate, and mixtures thereof;

(b) adding an encapsulatable material to the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (a); and

(c) denaturing the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (b) to precipitate the protein and thereby encapsulate the encapsulatable material in the protein.

The present invention is further directed to an encapsulated material which comprises a core having an encapsulatable material and a coating layer over the core having a protein having a mixture of hydrophobic and hydrophilic properties prepared by a process comprising the steps of:

(a) preparing an aqueous mixture of a protein having a mixture of hydrophobic and hydrophilic properties selected from the group consisting of isolated soy protein, whey protein isolate, caseinate, and mixtures thereof;

(b) adding an encapsulatable material to the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (a); and

(c) denaturing the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (b) to precipitate the protein and thereby encapsulate the encapsulatable material in the protein.

Detailed Description of the Invention

Applicant has found that a water-soluble encapsulatable material can be rendered water-insoluble, or substantially water-insoluble, by coating the encapsulatable material with a protein having a mixture of hydrophobic and hydrophilic properties. The method for rendering the water-soluble encapsulatable material water-insoluble comprises first admixing the protein having a mixture of hydrophobic and hydrophilic properties with water to form an aqueous mixture, wherein the protein may be partly in solution and partly in suspension, depending upon the exact composition of the protein and whether the protein is a single component or a mixture of components. The encapsulatable material is then added to the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties. Applicant believes that the hydrophilic portion of the protein helps solubilize the protein in water while the hydrophobic portion of the protein enables the protein to complex with and bind to the encapsulatable material. The protein is then denatured such as by lowering the pH of the mixture to below the isoelectric point of the protein, heating the mixture, or spray drying the mixture. During denaturation, applicant believes that the hydrophobic portion of the protein envelopes the water-soluble encapsulatable material rendering the encapsulatable material water-insoluble, or substantially water-insoluble.

In accord with the present invention, applicants have discovered a method for encapsulating an encapsulatable material with a protein having a mixture of hydrophobic and hydrophilic properties suitable for rendering a water-soluble color water-insoluble. The encapsulated material comprises (a) a core comprising an encapsulatable material; and (b) a coating layer over the core comprising a protein having a mixture of hydrophobic and hydrophilic

properties selected from the group consisting of isolated soy proteins, caseinates, whey proteins, and mixtures thereof. The method for encapsulating an encapsulatable material with a protein having a mixture of hydrophobic and hydrophilic properties comprises the steps of (a) preparing an aqueous mixture of a protein having a mixture of hydrophobic and hydrophilic properties; (b) adding an encapsulatable material to the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (a); and (c) acidifying the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (b) to precipitate the protein having a mixture of hydrophobic and hydrophilic properties and thereby encapsulate the encapsulatable material in the protein having a mixture of hydrophobic and hydrophilic properties. The present invention is further directed to the novel encapsulated material prepared by the novel process.

The proteins having a mixture of hydrophobic and hydrophilic properties present in the coating layer of the encapsulated material of the present invention include, but are not limited to, isolated soy protein, concentrated soy protein, whey protein isolate, caseinates, and mixtures thereof. In a preferred embodiment, the protein having a mixture of hydrophobic and hydrophilic properties is soy protein, preferably isolated soy protein or concentrated soy protein, and more preferably, isolated soy protein. One particularly preferred isolated soy protein is available from Protein Technologies Inc. of St. Louis, MO under the tradename FXP 920. FXP 920 has a typical amino acid profile, by weight, of 4.3% alanine, 7.6% arginine, 11.6% aspartic acid, 1.3% cysteine, 19.1% glutamic acid, 4.2% glycine, 2.6% histidine, 4.9% isoleucine, 8.2% leucine, 6.3% lysine, 1.3% methionine, 5.2% phenylalanine, 5.1% proline, 5.2% serine, 3.8% threonine, 1.3% tryptophan, 3.8% tyrosine, and 5.0% valine. This amino acid composition provides total sulfur amino acids of 2.6% and total aromatic amino acids of 9.0%. Histidine, isoleucine, leucine, lysine, methionine, phenylalanine, threonine, tryptophan, and valine are essential amino acids. The isolated soy protein has a chemical analysis of 91.0% protein on a dry basis, 5.0% moisture, less than 0.2% fat, less than 0.2% crude fiber, and 4.5% ash.

In another preferred embodiment, the protein having a mixture of hydrophobic and hydrophilic properties is whey protein, preferably whey protein isolate. Particularly preferred whey protein isolates are available from Land O'Lakes, Inc., Food Ingredients Division, of Minneapolis, MN under the tradename POWERPRO™ WHEY PROTEIN and from New Zealand Milk

Products, Inc. of Santa Rosa, CA under the tradename ALACEN 896, whey protein isolate. POWERPRO™ WHEY PROTEIN has a typical amino acid profile, by weight, of 4.9% alanine, 1.5% arginine, 11.7% aspartic acid, 2.1% cystine and cysteine, 19.5% glutamic acid, 1.2% glycine, 1.2% histidine, 5.8% isoleucine, 11% leucine, 9.4% lysine, 1.5% methionine, 2.7% phenylalanine, 6.2% proline, 5.1% serine, 7.2% threonine, 1.3% tryptophan, 2.5% tyrosine, and 5.3% valine.

Whey proteins in solution display a strong affinity for dyes as evidenced by the removal of dyes from the aqueous phase when the protein is rendered insoluble. The hydrophobicity and negative charge of some dyes appears to play a role on the effectiveness of the dye-protein complexing mechanism. Changes in the structural phases of the protein (denaturation, polymerization, coagulation, precipitation) lead to substantial alteration of the complexing/bonding process. Color bonding can survive thermal stress and acidic conditions by inducing optimal protein reactivity. The protein to dye ratio influences the effectiveness of dye bonding. The secondary (pasteurization, retorting, etc.) processing of foodstuff containing whey protein bonded color affects the migratory ability of the dye. Precipitated whey protein (heat, heat and acid, heat and Calcium, etc.) maintains the bonding with the dye. Complexed protein is suitable for spray drying processing. Color complexing takes place even if the protein and the dye powders are individually added to the ingredients being processed to make the matrix gel.

The amount of protein having a mixture of hydrophobic and hydrophilic properties used in the coating layer of the encapsulated material of the present invention is an amount effective to form the particular encapsulated material and may vary depending upon the particular protein having a mixture of hydrophobic and hydrophilic properties and encapsulatable material employed. In general, the amount of protein having a mixture of hydrophobic and hydrophilic properties present is the ordinary dosage required to obtain the desired result. In a preferred embodiment, the protein having a mixture of hydrophobic and hydrophilic properties in the coating layer of the encapsulated material of the present invention is present in an amount from about 0.01% to about 50%, preferably in an amount from about 0.05% to about 5%, and more preferably in an amount from about 0.1% to about 1%, by weight of the encapsulated material.

The encapsulatable material in the core of the encapsulated material of the present invention may be any water-soluble material that is desired to be rendered water-insoluble, or substantially water-insoluble. Preferably, the encapsulatable material is a water-soluble color. More preferably, the encapsulatable material is a water-soluble color selected from the group consisting of blue no. 1, red no. 3, red no. 40, yellow no. 4, yellow no. 5, green no. 3, and mixtures thereof.

The amount of encapsulatable material used in the core of the encapsulated material of the present invention is an amount effective to form the particular encapsulated material and may vary depending upon the particular protein having a mixture of hydrophobic and hydrophilic properties and encapsulatable material employed. In general, the amount of encapsulatable material present is the ordinary dosage required to obtain the desired result. In a preferred embodiment, the encapsulatable material in the core of the encapsulated material of the present invention is present in an amount from about 50% to about 99.99%, preferably in an amount from about 95% to about 99.95%, and more preferably in an amount from about 99% to about 99.9%, by weight of the encapsulated material.

The encapsulatable material in the core of the encapsulated material of the present invention may further comprise a hydrophobic compound to further coat the water-soluble material and render it water-insoluble. Preferably, the hydrophobic compound is selected from the group consisting of essential oils, compounded oil soluble flavors, vegetable oils, and mixtures thereof. Suitable essential oils include, but are not limited to, lime oil, lemon oil, orange oil, and the like, as well as spice oils such as nutmeg, cinnamon, pepper, and ginger and mixtures thereof. Suitable compounded oil soluble flavors include, but are not limited to, fixed flavors such as strawberry, blueberry, and grape. Suitable vegetable oils include, but are not limited to, coconut oil, corn oil, cottonseed oil, peanut oil, and soybean oil.

The amount of hydrophobic compound used in the core of the encapsulated material of the present invention is an amount effective to form the particular encapsulated material and may vary depending upon the particular hydrophobic compound, protein having a mixture of hydrophobic and hydrophilic properties, and encapsulatable material employed. In general, the amount of hydrophobic compound present is the ordinary dosage required to obtain the desired result. In a preferred embodiment, the hydrophobic

compound in the core of the encapsulated material of the present invention is present in an amount from about 0.1% to about 5%, preferably in an amount from about 0.5% to about 10%, and more preferably in an amount from about 1% to about 5%, by weight of the encapsulated material.

The present invention extends to a method for encapsulating an encapsulatable material with a protein having a mixture of hydrophobic and hydrophilic properties. In general, the method comprises the steps of (a) preparing an aqueous mixture of a protein having a mixture of hydrophobic and hydrophilic properties; (b) adding an encapsulatable material to the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (a); and (c) acidifying the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (b) to precipitate the protein having a mixture of hydrophobic and hydrophilic properties and thereby encapsulate the encapsulatable material in the protein having a mixture of hydrophobic and hydrophilic properties.

The first step in carrying out the method of the invention is to provide a solution of a protein having a mixture of hydrophobic and hydrophilic properties. The protein having a mixture of hydrophobic and hydrophilic properties is dissolved in water in an amount from about 2.5% to about 30%, by weight, preferably from about 8% to about 10% protein. The water is potable and may be treated or de-ionized prior to use.

The protein having a mixture of hydrophobic and hydrophilic properties solution is heated to a temperature between about 120°F and 180°F, preferably between about 160°F and 170°F in order to hydrate the protein and expose the hydrophobic regions. The heated solution is then cooled to between about 35°F and 100°F, preferably to between about 60°F and 70°F prior to use.

Between about 0.05% and about 0.5% by weight of an encapsulatable material, such as a water-soluble color, and optionally an essential oil, compounded oil soluble flavor, or vegetable oil, preferably between about 1% and 5% by weight, is added to the cooled protein having a mixture of hydrophobic and hydrophilic properties solution and preferably homogenized to form an emulsion. Water-soluble colors can be added to the emulsion in amounts between about 0.1% and 1.0% by weight, preferably in

amounts between about 0.4% and 0.7% by weight depending on the strength of the color desired.

The pH of the emulsion is then lowered to a pH below the isoelectric point of the protein having a mixture of hydrophobic and hydrophilic properties using a suitable food grade acid. For example, the isoelectric point of the preferred soy protein is between about 4.8 and 4.9. Accordingly, it is desirable to lower the pH of the emulsion to between about 4.0 and 4.6, preferably about 4.2.

Suitable acids include, but are not limited to, citric acid, phosphoric acid, ascorbic acid, malic acid, and mixtures thereof. In a preferred embodiment, citric acid is used.

Lowering the pH of the emulsion to below the isoelectric point of the protein having a mixture of hydrophobic and hydrophilic properties has the effect of precipitating the protein having a mixture of hydrophobic and hydrophilic properties into discrete oil droplets which encapsulate the encapsulatable material, thereby rendering the encapsulatable material insoluble in water.

The present invention also extends to an encapsulated material which comprises a core having an encapsulatable material and a coating layer over the core having a protein having a mixture of hydrophobic and hydrophilic properties prepared by a process comprising the steps of:

(a) preparing an aqueous mixture of a protein having a mixture of hydrophobic and hydrophilic properties;

(b) adding an encapsulatable material to the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (a); and,

(c) acidifying the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (b) to precipitate the protein having a mixture of hydrophobic and hydrophilic properties and thereby encapsulate the encapsulatable material in the protein having a mixture of hydrophobic and hydrophilic properties.

The colored and/or flavored emulsion may be used as a liquid emulsion or spray dried. Once the emulsion is spray dried, a colored and/or flavored free flowing powder is obtained. The spray dried powder can be used

for any desired application and the color and/or flavor will remain encapsulated in the protein.

The invention has application in texturally modified beverages where the ability to color a discrete portion of the beverage is important. Colors protected by the encapsulation technique of the invention are water-insoluble like lake colors. However, unlike lake colors, encapsulated colors produced by the method of the invention are pH stable.

For example, the jelled substances used in texturally modified beverages are disclosed in United States patent application serial no. 08/383,083. As disclosed therein, such substances can be prepared by mixing water and a suitable gelling agent to form a liquid composition. The liquid composition can be jelled by heating and then cooling. Alternatively, the liquid composition can be jelled by reacting the composition with mono- or di-valent ions such as calcium or sodium ions.

The water used to prepare such substances must be potable and is present in an amount between about 80% and 99.8%. by weight of the substance. It may be treated or deionized prior to use.

When a gelling agent is employed, the gelling agent may be gellan gum, xanthan gum, locust bean gum, pectin, alginates, carrageenans, starches, gelatin, and mixtures thereof. The gelling agent is used in an amount between about 0.1% and 5% by weight of the substance, preferably between about 0.2% and 1% by weight. In a preferred embodiment, gellan gum is used in an amount between about 0.1% and 0.5% by weight of the substance, preferably between about 0.2% and 0.4%, by weight.

A preferred gelling agent is a gellan gum. Gums, also called hydrocolloids, are polysaccharides which are polymers of sugar building blocks. Gums function as thickeners, stabilizers, suspending agents, gelling agents, film formers, aerating agents, flocculants, binders, emulsifiers, lubricants, and texturing and structuring agents. Gums are frequently classified as either thickening agents or gelling agents. Typical thickening agents include starches, guar gum, locust bean gum, xanthan gum, gum arabic, carboxymethylcellulose (CMC), alginates, methylcellulose, gum karaya and gum tragacanth. The major gelling agents are gelatin, starch, alginate, pectin, carrageenan, agar and methylcellulose. Some gums, such as alginates, can function both as a

thickening agent and a gelling agent. Certain thickening agents, like xanthan gum and locust bean gum, can form gels when used together. The term "gelling agent" refers to the ability of a gum to convert water from a flowable liquid to a demoldable solid or gel. Gels are commonly formed by cooling a hot solution of a gelling agent or by introducing a gel-forming agent, typically metal ions, into a gelling solution. The transition from the liquid state to the gelled state occurs by controlled association of the gum molecules to form a three-dimensional network in which the water is entrapped. Although the hydration of thickening agents or gelling agents is generally referred to as "solution preparation", polysaccharides actually form hydrocolloidal dispersions rather than true solutions.

Gellan gums are fermentation hydrocolloids produced by the microorganism *Pseudomonas elodea* and manufactured by Kelco Corp., San Diego, California. The constituent sugars of gellan gum are glucose, glucuronic acid, and rhamnose in the molar ratio of 2:1:1, respectively. These sugars are linked together to give a primary structure consisting of a linear tetrasaccharide repeating unit. In the native high acyl form, gellan gum contains two acyl substituents, acetate and glycerate. Both substituents are located on the same glucose residue, and on average, there is one glycerate per repeat and one acetate for every two repeats. The acyl groups have a profound influence on gel characteristics.

Preferred gellan gums are K9A50, a nonclarified form of gellan gum for industrial use, KELCOGEL™, a low acyl gellan gum for foods and industrial products, and GELRITE™, a low acyl gellan gum for microbiological media, plant tissue culture, and pharmaceutical applications. Preferably, the gellan gum is a low acyl gellan gum such as KELCOGEL™ gellan gum or GELRITE™ gellan gum, gellan gums in which the acyl groups are removed completely. More preferably, the gellan gum is KELCOGEL™. These gellan gums are described in more detail in United States patent no. 4,326,053 and United States patent no. 4,326,052, which disclosures are incorporated herein by reference.

The amount of gellan gum used in a texturally modified beverage is an amount effective to form the particular gel and may vary depending upon the dosage recommended or permitted for the particular gellan gum. In general, the amount of gellan gum present is the ordinary dosage required to obtain the desired result. In a preferred embodiment, the gellan gum in a texturally

modified beverage is present in an amount from about 0.1% to about 1.5%, preferably in an amount from about 0.1% to about 1%, and more preferably in an amount from about 0.1% to about 1.5%, by weight.

Other gums may optionally be incorporated into a texturally modified beverage. Nonlimiting examples of such other gums include xanthan gum, carrageenan, pectin, alginate, carboxymethylcellulose, and the like, and mixtures thereof. These optional gums may be used in amounts from about 0.02% to about 0.5%, and preferably from about 0.025% to about 0.2%, by weight.

Other water-soluble components that may be included in a texturally modified beverage include, for example, sweeteners, acids, ion providers or buffers, sequestrants, preservatives, and the like. The texture of the jelled substance is a function of the components of the substance and their proportions and is measured using standards of elasticity, brittleness, hardness, modulus, and cohesiveness.

For example, carbohydrate sweeteners may be used to aid in dispersion of the gelling agent. In addition, the sweetener can also weight the jelled substance so that pieces of the substance do not float when incorporated into a liquid composition.

When sweeteners are utilized, the present invention contemplates the inclusion of those sweeteners well known in the art, including both natural and artificial sweeteners. Thus, sweeteners may be chosen from the following non-limiting list: sugars such as sucrose, glucose (corn syrup), dextrose, invert sugar, fructose, and mixtures thereof; saccharine and its various salts such as the sodium or calcium salt; cyclamic acid and its various salts such as the sodium salt; the dipeptide sweeteners such as aspartame; dihydrochalcone; Stevia rebaudiana (Steviodide); and sugar alcohols such as sorbitol, sobitol syrup, mannitol, xylitol, and the like. Also contemplated as an additional sweetener is the nonfermentable sugar substitute (hydrogenated starch hydrolysates) which is described in U.S. Reissue Pat. No. 26,959. Also contemplated is the synthetic sweetener 3,6-dihydro-6-methyl-1-1,2,3-oxathiazin-4-one-2,2-dioxide particularly the potassium (Acesulfame-K), sodium and calcium salts thereof as described in German Patent No. 2,001,017.7.

The sweetener can be used in solid or in dissolved form and, when used, is generally present in an amount between about 0.1% and 20% by weight of the substance depending on the physical properties of the jelled substance that are desired.

When sweetening agents (sweeteners) are used, those sweeteners well known in the art, including both natural and artificial sweeteners, may be employed. The sweetening agent used may be selected from a wide range of materials including water-soluble sweetening agents, water-soluble artificial sweetening agents, water-soluble sweetening agents derived from naturally occurring water-soluble sweetening agents, dipeptide based sweetening agents, and protein based sweetening agents, including mixtures thereof. Without being limited to particular sweetening agents, representative categories and examples include:

(a) water-soluble sweetening agents such as monosaccharides, disaccharides and polysaccharides such as xylose, ribose, glucose (dextrose), mannose, galactose, fructose (levulose), sucrose (sugar), maltose, invert sugar (a mixture of fructose and glucose derived from sucrose), partially hydrolyzed starch, corn syrup solids, dihydrochalcones, monellin, steviosides, and glycyrrhizin, and mixtures thereof;

(b) water-soluble artificial sweeteners such as soluble saccharin salts, i.e., sodium or calcium saccharin salts, cyclamate salts, the sodium, ammonium or calcium salt of 3,4-dihydro-6-methyl-1,2,3-oxathiazine-4-one-2,2-dioxide, the potassium salt of 3,4-dihydro-6-methyl-1,2,3-oxathiazine-4-one-2,2-dioxide (Acesulfame-K), the free acid form of saccharin, and the like;

(c) dipeptide based sweeteners, such as L-aspartic acid derived sweeteners, such as L-aspartyl-L-phenylalanine methyl ester (Aspartame) and materials described in United States patent no. 3,492,131, L-alpha-aspartyl-N-(2,2,4,4-tetramethyl-3-thietanyl)-D-alanin-amide hydrate (Alitame), methyl esters of L-aspartyl-L-phenylglycerine and L-aspartyl-L-2,5-dihydrophenyl-glycine, L-aspartyl-2,5-dihydro-L-phenylalanine; L-aspartyl-L-(1-cyclohexen)-alanine, and the like;

(d) water-soluble sweeteners derived from naturally occurring water-soluble sweeteners, such as chlorinated derivatives of ordinary sugar (sucrose), e.g., chlorodeoxysugar derivatives such as derivatives of chlorodeoxysucrose or chlorodeoxygalactosucrose, known, for example, under the product designation of Sucralose; examples of chlorodeoxysucrose and chlorodeoxygalacto-sucrose derivatives include but are not limited to: 1-chloro-1'-deoxysucrose; 4-chloro-4-deoxy-alpha-D-galacto-pyranosyl-alpha-D-

fructofuranoside, or 4-chloro-4-deoxygalactosucrose; 4-chloro-4-deoxy-alpha-D-galacto-pyranosyl-1-chloro-1-deoxy-beta-D-fructo-furanoside, or 4,1'-dichloro-4,1'-dideoxygalactosucrose; 1',6'-dichloro-1',6'-dideoxysucrose; 4-chloro-4-deoxy-alpha-D-galacto-pyranosyl-1,6-dichloro-1,6-dideoxy-beta-D-fructo-furanoside, or 4,1',6'-trichloro-4,1',6'-trideoxygalacto-sucrose; 4,6-dichloro-4,6-dideoxy-alpha-D-galacto-pyranosyl-6-chloro-6-deoxy-beta-D-fructofuranoside, or 4,6,6'-trichloro-4,6,6'-trideoxygalactosucrose; 6,1',6'-trichloro-6,1',6'-trideoxysucrose; 4,6-dichloro-4,6-dideoxy-alpha-D-galacto-pyranosyl-1,6-dichloro-1,6-di-deoxy-beta-D-fructofuranoside, or 4,6,1',6'-tetrachloro-4,6,1',6'-tetradeoxygalacto-sucrose; and 4,6,1',6'-tetrachloro-4,6,1',6'-tetradeoxy-sucrose; and

(e) protein based sweeteners such as thaumaoccous danielli (Thaumatin I and II).

In general, an effective amount of sweetening agent is utilized to provide the level of sweetness desired in the particular oral topical antiseptic composition, and this amount will vary with the sweetener selected and the final oral antiseptic product desired. The amount of sweetener normally present is in the range from about 0.0025% to about 90%, by weight of the oral topical antiseptic composition, depending upon the sweetener used. The exact range of amounts for each type of sweetener is well known in the art and is not the subject of the present invention.

Acids can be selected from food grade organic acids. For example, citric acid, malic acid, tartaric acid, fumaric acid, lactic acid, and mixtures thereof can be used. When used, the acid is present in an amount between about 0.05% and 0.5% by weight of the substance, preferably between about 0.1% and 0.25% by weight.

A buffer can be selected from food approved buffering agents including, but not limited to, sodium citrate, potassium citrate, sodium tripolyphosphate, sodium hexametaphosphate, and mixtures thereof. The buffer is used in an amount between about 0.02% and 0.2% by weight of the substance, preferably between about 0.05% and 0.1%, by weight.

Sequestrating agents are used when necessary to correct ion imbalances that may be present in the water. Such an ion imbalance can cause difficulties with gellation such as a failure of the liquid composition including water and gelling agent to gel or premature gelling of the composition.

A preservative such as sodium benzoate, potassium sorbate, and mixtures thereof is optionally used in the substance. When used, the preservative is present in an amount up to 0.1%, by weight.

In addition, an encapsulated color and/or flavor prepared in accordance with the encapsulation method of the invention is used to color and/or flavor the jelled substance. The encapsulated color may be used in amounts between about 0.1% and 5.0% based on the intensity of the color desired and may be used in wet or spray dried form.

To prepare the jelled substances in accordance with the invention, all of the dry materials are blended and added to the water. The water and dry materials are stirred until a homogeneous dispersion is formed. The temperature of the dispersion is raised to between about 160° and 190°F and maintained at that temperature until the gum is fully hydrated. The solution will be clear when the gum is fully hydrated. The solution is then cooled to between about 120° and 150°F and the encapsulated color and/or flavor is added in either liquid emulsion or spray dried form.

In a preferred embodiment, the resulting liquid composition is gelled by contacting the composition with a solution of a mono- or di-valent salt such a sodium chloride or calcium chloride. When the composition is contacted dropwise with the salt solution, beads of the jelled substance are formed.

In an alternate embodiment, the composition is gelled by contacting the composition with a strong acid such as a 2% solution of citric acid in water. In a further alternate embodiment, the composition is gelled by allowing it to cool and set in a mold. In this embodiment, the jelled substance can be cut into any desired shape by conventional means that are well known to those skilled in the art.

The jelled substance can be preserved by the addition of a preservative such as sodium benzoate. The encapsulated color provides an intensely vibrant color to the jelled substance although the precise degree of color intensity is, of course, determined by the amount of color that is used.

The pieces or beads of the jelled substance can be incorporated in a liquid composition having a pH between about 2.5 and 6 in an amount

between about 1% and 10% by weight of the composition, preferably in an amount between about 5% and 8% by weight. The liquid composition can be hot or cold, carbonated or non-carbonated, alcoholic or non-alcoholic, caffeinated or noncaffeinated, clear or cloudy. Suitable liquid compositions include, but are not limited to, soda, coffee, tea, beer, milk, mouthwash, and the like. The jelled substance remains discrete and retains its color during and subsequent to processing of the liquid composition.

The present invention is further illustrated by the following examples which are not intended to limit the effective scope of the claims. All parts and percentages in the examples and throughout the specification and claims are by weight of the final composition unless otherwise specified.

Example 1

Ninety (90) parts of water and 10 parts of FXP 920 were combined and heated to 185°F to form a 10% isolated soy protein solution. The solution was cooled to 80°F. Eighty-five grams (85g) of the 10% isolated soy protein solution were emulsified with 15g of a compounded oil soluble strawberry flavor. Fifteen hundredths of a gram (0.15g) of dissolved citric acid was added to the emulsion with shear to achieve a precipitate with a final pH of 4.7. The strawberry flavor was encapsulated in the soy protein.

Example 2

Eight-five grams (85g) of a 10% isolated soy protein solution prepared as described in Example 1 were emulsified with 15g of a compounded oil soluble coconut flavor. Six tenths of a gram (0.6g) of dissolved citric acid was added to the emulsion with shear to achieve a precipitate with a final pH of 4.82. The coconut flavor was encapsulated in the soy protein.

Example 3

Eighty-five grams (85g) of a 10% isolated soy protein solution prepared as described in Example 1 were emulsified with 15g of lime oil and 2g of a blend of blue no. 1 and Yellow no. 4. Three tenths of a gram (0.3g) of dissolved citric acid was added to the emulsion with shear to achieve a precipitate with a final pH of 4.7. The lime flavor and green color were encapsulated in the soy protein.

Example 4

Eight-five grams (85g) of a 10% isolated soy protein solution prepared as described in Example 1 were emulsified with 15g of a compounded oil soluble concord grape flavor and 1.25g of a mixture of 90% red no. 3 and 10% blue no. 1. The precipitate had a final pH of 4.82. Excellent color retention was achieved with virtually no leaching.

Example 5

Ninety (90) parts water and 10 parts FXP 920 were combined and heated to 185°F to form a 10% isolated soy protein solution. The solution was cooled to 80°F. Two hundred grams (200g) of the 10% isolated soy protein solution were emulsified with 10g of vegetable oil and 1.5g of a blend of blue no. 1 and yellow no. 4. Two grams (2.0g) of dissolved citric acid was added to the emulsion with shear to achieve a precipitate having a final pH of 4.4. The emerald green color was encapsulated in the soy protein.

Example 6

Eighty three and forty three hundredths (83.43) parts cold water were measured into a weighed glass beaker. A dry blend of 16 parts granulated sucrose, 0.3 parts gellan gum, 0.15 parts sodium citrate, and 0.12 parts xanthan gum was prepared and slowly added to the water with agitation. When all of the ingredients were suspended in the water, the temperature was raised to 180°F with gentle stirring.

The liquid was maintained at 180°F for about 10 minutes until visual inspection indicated that hydration of the gum was complete. The heat source was removed and the beaker was cooled in a cold water bath until a temperature of 150°F was achieved. One part encapsulated emerald green prepared in accordance with Example 5 were added and the mixture was stirred until complete dispersion of the color was achieved. The resultant green liquid was added dropwise into a calcium chloride solution and green beads were formed on contact of the liquid with the solution.

The resulting beads of green jelled substance were incorporated into a non-carbonated beverage at a level of 5%, by weight. The beverage was

bottled, capped, and pasteurized at 180°F for ten (10) minutes and cooled. The green beads exhibited excellent color retention with virtually no color leaching into the beverage.

Example 7

The following methods were employed to minimize the migration of FD&C colorants introduced into gelled matrixes that are required to undergo heat treatment (up to 250°F) under acid conditions (pH range 4 5 to 2.5).

a) Integral Processing

The gel matrix is processed by heating and adding Ca^{++} or acid and subsequently preserving the matrix under acid conditions (pH 3 to 4). The whey protein powder and the dye powder are added to the matrix dry mix and the matrix processing conditions are followed. The ratio of protein to dye is preestablished on the basis of the specific protein and dye. The ratio of dye to matrix ingredients is based on the coloring level desired and on the desired textural properties of the matrix. The complexed protein reaches a water-insoluble condition at the end of the matrix processing (preservation).

b) Unprecipitated Color Solution

This process requires a heat stable protein. The dye powder is added to the protein solution (typ. 1% to 50% w/w whey), the mass is then heated to 85°C to 95°C to induce denaturation and polymerization, cooled, and finally added to the matrix solution before matrix gelling. The complexed protein reaches a water-insoluble state at the end of the matrix processing.

c) Partially Precipitated Color Solution

The dye is added to the protein solution (typ. 1% to 25% w/w whey), acid is then added to induce optimal coagulation/precipitation during heating (typically, the cold solution should be at pH 4 to 5.5, depending on the mixing characteristics of the heating reactor). During heating to 70°C to 80°C (the maximum temperature is also based on the mixing method), the solution is well mixed to prevent lumping of coagulate and milled through a wet colloidal mill to control the particle size of the precipitate to below 50 microns. The

cooled solution containing the settled fine precipitate is added and mixed thoroughly to the matrix solution before gelling.

d) Dried Color

The color solutions prepared with method b) and c) can be dried and milled or spray dried and added as powder to the matrix solution before gelling. When the spray drying method is selected, the solution heating step can be eliminated when particular protein to dye ratios allow it.

Throughout this application, various publications have been referenced. The disclosures in these publications are incorporated herein by reference in order to more fully describe the state of the art.

The invention being thus described, it will be obvious that the same may be varied in many ways. Such variations are not to be regarded as a departure from the spirit and scope of the invention and all such modifications are intended to be included within the scope of the following claims.

We claim

1. An encapsulated material which comprises:

(a) a core comprising an encapsulatable material; and

(b) a coating layer over the core comprising a protein having a mixture of hydrophobic and hydrophilic properties selected from the group consisting of isolated soy protein, whey protein isolate, caseinate, and mixtures thereof.

2. The encapsulated material according to claim 1, wherein the protein having a mixture of hydrophobic and hydrophilic properties is isolated soy protein.

3. The encapsulated material according to claim 1, wherein the protein having a mixture of hydrophobic and hydrophilic properties is whey protein isolate.

4. The encapsulated material according to claim 1, wherein the encapsulatable material is a water-soluble color.

5. The encapsulated material according to claim 4, wherein the water-soluble color is selected from the group consisting of blue no. 1, red no. 3, red no. 40, yellow no. 4, yellow no. 5, green no. 3, and mixtures thereof.

6. The encapsulated material according to claim 1, wherein the encapsulatable material further comprises a compound selected from the group consisting of essential oils, compounded oil soluble flavors, vegetable oils, and mixtures thereof.

7. The encapsulated material according to claim 6, wherein the compound is an essential oil selected from the group consisting of lime oil, lemon oil, orange oil, nutmeg, cinnamon, pepper, ginger, and mixtures thereof.

8. A method for encapsulating an encapsulatable material with a protein having a mixture of hydrophobic and hydrophilic properties which comprises the steps of:

(a) preparing an aqueous mixture of a protein having a mixture of hydrophobic and hydrophilic properties selected from the group consisting of isolated soy protein, whey protein isolate, caseinate, and mixtures thereof;

(b) adding an encapsulatable material to the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (a); and

(c) denaturing the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (b) to precipitate the protein and thereby encapsulate the encapsulatable material in the protein.

9. The method according to claim 8, wherein the protein having a mixture of hydrophobic and hydrophilic properties is isolated soy protein.

10. The method according to claim 8, wherein the protein having a mixture of hydrophobic and hydrophilic properties is whey protein isolate.

11. The method according to claim 8, wherein the encapsulatable material is a water-soluble color.

12. The method according to claim 11, wherein the water-soluble color is selected from the group consisting of blue no. 1, red no. 3, red no. 40, yellow no. 4, yellow no. 5, green no. 3, and mixtures thereof.

13. The method according to claim 8, wherein the encapsulatable material further comprises a compound selected from the group consisting of essential oils, compounded oil soluble flavors, vegetable oils, and mixtures thereof.

14. The method according to claim 13, wherein the compound is an essential oil selected from the group consisting of lime oil, lemon oil, orange oil, nutmeg, cinnamon, pepper, ginger, and mixtures thereof.

15. The method according to claim 8, wherein the protein having a mixture of hydrophobic and hydrophilic properties is present in the aqueous mixture in step (a) in an amount from about 2.5% to about 30%, by weight.

16. The method according to claim 8, wherein the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties comprising an encapsulatable material in step (b) is homogenized to an emulsion, prior to the denaturation in step (c).

17. The method according to claim 8, wherein the aqueous mixture of protein in step (c) is denatured by acidifying the aqueous mixture of protein.

18. The method according to claim 17, wherein the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (c) is acidified to lower the pH of the emulsion to below the isoelectric point of the protein having a mixture of hydrophobic and hydrophilic properties.

19. The method according to claim 17, wherein the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (c) is acidified with an acid selected from the group consisting of citric acid, phosphoric acid, ascorbic acid, malic acid, and mixtures thereof.

20. The method according to claim 17, wherein the acid is citric acid.

21. The method according to claim 8, wherein the aqueous mixture of protein in step (c) is denatured by heating the aqueous mixture of protein.

22. The method according to claim 8, wherein the aqueous mixture of protein in step (c) is denatured by spray drying the aqueous mixture of protein.

23. An encapsulated material which comprises a core having an encapsulatable material and a coating layer over the core having a protein having a mixture of hydrophobic and hydrophilic properties prepared by a process comprising the steps of:

(a) preparing an aqueous mixture of a protein having a mixture of hydrophobic and hydrophilic properties selected from the group consisting of isolated soy protein, whey protein isolate, caseinate, and mixtures thereof;

(b) adding an encapsulatable material to the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (a); and

(c) denaturing the aqueous mixture of protein having a mixture of hydrophobic and hydrophilic properties in step (b) to precipitate the protein and thereby encapsulate the encapsulatable material in the protein.

24. The encapsulated material according to claim 23, wherein the protein having a mixture of hydrophobic and hydrophilic properties is isolated soy protein.

25. The encapsulated material according to claim 23, wherein the protein having a mixture of hydrophobic and hydrophilic properties is whey protein isolate.

26. The encapsulated material according to claim 23, wherein the encapsulatable material is a water-soluble color.

27. The encapsulated material according to claim 23, wherein the encapsulatable material further comprises a compound selected from the group consisting of essential oils, compounded oil soluble flavors, vegetable oils, and mixtures thereof.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US97/12245

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :A23L 1/221
US CL : 426/96

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 426/96, 98, 650, 651

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Please See Extra Sheet.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X ---- Y	US, A, 5,418,010 (JANDA ET AL) 23 May 1995 (23.05.95), see columns 2-5.	1-14,16-18,23-27 ---------- 15,19 20-22

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

*	Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A"	document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E"	earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search	Date of mailing of the international search report
18 AUGUST 1997	25 SEP 1997
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	Authorized officer LIEN TRAN for Nikki Thomas
Facsimile No. (703) 305-3230	Telephone No. (703) 308-0648

Form PCT/ISA/210 (second sheet)(July 1992)*

B. FIELDS SEARCHED
Electronic data bases consulted (Name of data base and where practicable terms used):

APS
search terms: essential oil, soluble flavor oil, water soluble color, soy protein, caseinate, whey protein, encapsulating, coating, precipitating